SANDIA NATIONAL LABORATORIES
CIVILIAN RADIOACTIVE WASTE MANAGEMENT
TECHNICAL PROCEDURE

TP-65

REVISION 04

DRYING GEOLOGICAL SAMPLES TO CONSTANT WEIGHT

Effective Date: 10/15/01

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(Reviewer signatures above serve to document the review and resolution of comments)

REVISION HISTORY

Revision	Description
A	Initial Issue
B	Minor Revisions
3	Revised to incorporate QAIP 20-1 requirements and make other minor improvements.
4	TP 65 was deactivated during Audit BSC-ARC-01-010. It is now reactivated for the work to be performed under TWP *Subsurface Performance Testing for License Application (LA) for Fiscal Year 2001, TWP-EBS-MD-000009 Rev. 03*. No major technical revisions were required from revision 3, only reference to current procedures have been revised (i. e., WAs to TWPs, QAIPs to APs) and other minor editorial revisions.

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1.0 Scope and Objective

This procedure applies to oven drying geological specimens to constant weight. The procedure is intended for implementation in a laboratory environment and will be used as needed in support of Sandia National Laboratories (SNL) work for the Yucca Mountain Site Characterization Project. The objective of this procedure is to define a process to oven dry geological specimens to constant weight while minimizing thermal shock. This procedure is based in part on the results of drying experiments run at SNL (see references in Section 7.0 of this procedure).

2.0 Prerequisites

Before performing this technical procedure, personnel must be trained by the Principal Investigator and demonstrate their proficiency in performing this procedure. The Principal Investigator has responsibility for generating a record of personnel proficiency training.

Personnel using this procedure are responsible for ensuring that a controlled copy of this procedure is available and used for performing this procedure.

3.0 Description of Activity

Test specimens may be dried in a ventilated oven, at a specified relative humidity (RH), or with a vacuum applied. A sequence of weighing, heating, and cooling is repeated until a specimen reaches constant weight.

4.0 Operations

4.1 Specimen Identification and Chain-of-Custody Activities

4.1.1 Specimen Identification (ID)

Samples will be maintained in accordance with QAIP 20-03, *Sample Control.* Specimen IDs will appear on the specimens or containers, and corresponding specimen Chain-of Custody Forms (QAIP 20-03, App. B) and will be used throughout work using this TP. If a specimen ID marking becomes illegible, it should be written on a portion of the specimen where it would not be removed during any subsequent process. In any case, only one unmarked specimen should be outside of its container at any one time.

4.1.2 Documents Requirements

Responsibility for ensuring that work is performed and documented in accordance with this procedure lies with the Principal Investigator.

4.2 Equipment

The following are necessary to oven dry a sample:

4.2.1 Ventilated (or vacuum, or controlled humidity) oven. The oven must: (1) be capable of the required temperature range, (2) have heating and cooling rates controlled to $\leq 2\ ^{o}C$/min, (3) have temperature stability within $\pm 5\ ^{o}C$ at the minimum and maximum temperature hold points, and (4) be capable of housing a temperature-indicating device for verifying heating and cooling rates and hold-point temperatures.

4.2.2 Temperature-indicating and recording device for measuring oven temperatures. The recording device must be capable of providing electronic or hard copy documentation of temperature (^{o}C) versus time during the time that the oven is heating or cooling and at all temperature hold points. The accuracy of the temperature-indicating and recording devices should be within 2 ^{o}C for temperatures between 25 ^{o}C and the drying temperature.

4.2.3 Balance. A balance having a range greater than the initial weight of the sample with an accuracy greater than or equal to 0.01% of the predried sample weight. For specimens of low mass (<50 g), this accuracy may not be feasible. An accuracy of 0.02% of the predried weight may be used if approved by the PI. This change may be documented by modifying, initialing and dating Part 2 of the Oven Drying Data Sheet (ODDS; Appendix A).

The manufacturer/make, model, serial number, date of last calibration, and calibration due date of the balance will be documented on the ODDS. Calibration of the oven temperature-indication and recording device will be documented in the test records. If make, model, or serial number are not available, an identifier will be defined by the user of this procedure according to AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards.*

4.2.4 Vacuum pump or connection to house vacuum and vacuum gauge (or corresponding pressure indicator). If used, any unit of pressure may be used. The specifications (manufactures, make, model, serial numbers) for the equipment used must be noted on the ODDSs . The gauge or corresponding pressure indicator may be marked with the notation "FOR INDICATION ONLY"and is not required to follow AP-12.1Q.

4.2.5 Hygrometer. If drying is performed at constant relative humidity (RH), a hygrometer is needed. The specifications must be documented in the test records. The hygrometer may be marked with the notation "For Indication Only."

4.3 Oven-Drying Procedure

4.3.1 Verify that the following prerequisites have been met:

- The most current revision of this TP is being used.
- The sample identification and sample Chain-of-Custody requirements given in Section 4.1 have been met.
- The balance has been warmed up and leveled in accordance with the owner's manual, and calibrations are current.

4.3.2 The Oven-Drying Data Sheet (ODDS)

The Oven-Drying Data Sheet provides documentation of the drying procedure. The forms given in Appendix A may be used, or other sheets may be generated if all required information is recorded. If ODDS are used, then data recording will consist of: (1) one ODDS Part 1, (2) as many Part 1 continuation sheets as needed, (3) one ODDS Part 2, and (4) as many Part 2 continuation sheets as needed. There will be a 1:1 correspondence between ODDS Part 1 and ODDS Part 2 data sheets in the test records

4.3.2 The following information should be recorded on the first page of the ODDS Part 1:

- Technical Workplan (TWP) identification.
- Specimen identification (ID).

- Make/manufacturer, model, serial number date of last calibration and calibration due date of balance.
- Make/manufacturer, model, serial number date of last calibration and calibration due date of the temperature recording device.
- Nominal heating and cooling rates.

4.3.4 Record the specimen ID on ODDS Part 1 of continuation pages and on ODDS Part 2 data sheet(s).ODDS Part 2 data sheets will also include the initial specimen mass (before drying) or the specimen mass after the last drying cycle (continuation sheets).

4.3.5 Step-by-Step Operating Procedures

NOTE 1: More than one sample may be dried at one time.

1. Place the specimen(s) to be dried in the oven. The temperature in the oven should be between ambient and 40 ^{o}C. Apply vacuum, if applicable.

2. Record the date, time, and temperature of the oven at the time the sample is placed in the oven (ODDS Part 1).

3. Heat at $\leq$ 2 ^{o}C/min until the oven reaches peak temperature. The specimen can be dried at any temperature, depending upon the application. If the specimen contains clays and zeolites that should not be dehydrated, then dry at 60 ^{o}C and 45% RH (Bush & Jenkins, 1970). A standard temperature for drying (non-humidified) is 110 $\pm$ 5 ^{o}C.

4. Record the date, time and temperature when the drying cycle begins, which is when the oven reaches peak temperature (ODDS Part 1).

5. Maintain the sample at peak temperature; at least 120 hours is recommended. A shorter time may be specified by the PI and documented in the test records. ASTM D2216 states that in most cases 12-16 hours is sufficient. Monitor the oven temperature at regular intervals during this time, using the recording device specified in Section 4.2.2. In the test records, note any time periods and/or oven temperatures that fall outside of this range. If vacuum or RH was applied, record the pressure or RH and then return the conditions in the oven to ambient.

6. Record the date, time, and temperature of the oven when the drying cycle ends (ODDS Part 1). If vacuum was used, then vent to atmosphere or backfill with dry nitrogen gas.

7. Cool the oven at $\leq$ 2 ^{o}C/min until the oven temperature is between ambient and 40 ^{o}C.

8. Record the date, time, and temperature of the oven when the oven reaches the cooling temperature (ODDS Part 1).

9. Remove the sample from the drying oven and weigh it. Record all weights to the maximum resolution of the balance (ODDS, Part 2). A sample should be weighed within 15 min of its removal from the oven.

10. Calculate the weight change for each specimen, i.e., the change in specimen weight occurring over the last oven-drying cycle and record (ODDS Part 2).

    The percent mass change of a specimen is calculated by dividing the change in sample mass by the mean specimen mass measured immediately before the most recent oven drying. If the

percent mass change is within ± 0.05%, the specimen has met the specification and oven drying of the specimen is terminated (go to Step 11). If the percent mass change is greater than ± 0.05%, the specimen has not met the specification, return to Step 1. ASTM D2216 used a stability criterion of 0.1%. The ASTM criterion can be used if documented in the test records by the PI (ODDS Part 2 comments section). An example of the need to change this criteria would be if the dry mass value is difficult to reproduce because of specimen rehydration. If the drying process is terminated without meeting either of these criteria, then this must be clearly documented.

11. The following should be done for each specimen meeting the specification.

    - Remove the specimen from the drying oven.
    - Sequentially number the ODDS used for each sample, e.g., Page 1 of 2, Page 2 of 2.
    - Verify that Parts 1 and 2 of the ODDS are complete.

4.4 Postrequisites

When the last sample has been removed from the oven, verify that the oven has been turned off and returned to ambient temperature and pressure.

4.5 Safety

Because samples are not to be handled at temperatures above 40^{o} C, there should be no safety hazards other than normal hazards of the equipment. Operations will be in accordance with safety requirements of the facility where the work is being performed and those of the employer of person(s) performing the work.

5.0 Nonconformance, Deviations and Corrective Actions

Any nonconformances or deviations must be reported to the Principal Investigator as soon as possible. Deviations, deficiencies, and corrective actions must be determined and documented in accordance with AP-15.2Q, *Control of Nonconformances* and AP-16.1Q, *Management of Conditions Adverse to Quality*.

6.0 QA Records

QA records, and any corrections or changes thereto, generated as a result of implementing this procedure will be prepared and submitted as inclusionary QA records (QA:QA) by the Principal Investigator in accordance with AP-17.1Q, *Record Source Responsibilities for Inclusionary Records.* These records include:

- Proficiency Training records (Section 2.0)
- Oven Drying Data Sheets (ODDS) Parts 1 and 2 (Section 4.3) or equivalent information recorded on different forms.
- Calibration records (if applicable Section 4.2)

7.0 References

Memo to F.B. Nimick, 6313, from B.M. Schwartz, 6313, “Determination of Drying Procedures for Densely Welded Busted Butte NX Size Test Specimens,” August 8, 1983.

Memo to F.B. Nimick, 6313, from B.M. Schwartz, 6313, "Drying Procedure for NX Size or Smaller Samples," March 30, 1984.

ASTM D2216-92, "Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock."

AP-SIII.1Q, *Scientific Notebooks* (if applicable)

AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*

AP-15.2Q, *Control of Nonconformances*

AP-16.1Q, *Management of Conditions Adverse to Quality*

AP-17.1Q, *Records Source Responsibilities for Inclusionary Record*

QAIP 20-03, *Sample Control*

APPENDIX A

OVEN DRYING DATA SHEET (Part 1)

TWP No.: _____ Specimen ID: _____
Make/manufacturer/model/serial #
Of balance: _____
Of data recording device: _____
Date of last calibration of balance:_____ Due Date: _____
Date of last calibration of data recorder:_____ Due Date: _____
Heating Rate: _____ Cooling Rate: _____

		Begin Heating	End Heating	Begin Cooling	End Cooling	Pressure and RH (optional)
Oven Cycle 1	Date					
	Time					
	Temp (°C)					
Oven Cycle 2	Date					
	Time					
	Temp (°C)					
Oven Cycle 3	Date					
	Time					
	Temp (°C)					
Oven Cycle 4	Date					
	Time					
	Temp (°C)					
Oven Cycle 5	Date					
	Time					
	Temp (°C)					

Comments: _____

Work performed by (print, sign, and date): _____
Location of Work: Sandia National Laboratories

OVEN DRYING DATA SHEET (Part 1, concluded)

Specimen ID: _____

		Begin Heating	End Heating	Begin Cooling	End Cooling	Pressure and RH (optional)
Oven Cycle #____	Date					
	Time					
	Temp (°C)					
Oven Cycle #____	Date					
	Time					
	Temp (°C)					
Oven Cycle #____	Date					
	Time					
	Temp (°C)					
Oven Cycle #____	Date					
	Time					
	Temp (°C)					
Oven Cycle #____	Date					
	Time					
	Temp (°C)					

Comments: _____
_____
_____

Work performed by (print, sign, and date): _____
Location of Work: Sandia National Laboratories

OVEN DRYING DATA SHEET (Part 2)

Specimen ID: _____
Specimen mass before oven-drying: _____ (g)

	Mass After Drying, (g)	Change in Mass[(a)] (%)	Change Within Specifications? (< ± 0.05%) (Y/N)
Oven Cycle #1			
Oven Cycle #2			
Oven Cycle #3			
Oven Cycle #4			
Oven Cycle #5			

(a) 100 × (Final Mass - Initial Mass) / Initial Mass

Comments: _____
_____
_____

Work performed by (print, sign, and date): _____
Location of Work: Sandia National Laboratories

OVEN DRYING DATA SHEET (Part 2, concluded)

Specimen ID: _____
Specimen mass after last oven-drying cycle: _____ (g)

	Mass After Drying, (g)	Change in Mass[(a)] (%)	Change Within Specifications? (< ± 0.05%) (Y/N)
Oven Cycle # _____			
Oven Cycle # _____			
Oven Cycle # _____			
Oven Cycle # _____			
Oven Cycle # _____			

(a) 100 × (Final Mass - Initial Mass) / Initial Mass

Comments: _____
_____
_____

Work performed by (print, sign, and date): _____
Location of Work: Sandia National Laboratories